Claim 12 has been amended to more particularly point out and distinctly claim that which Applicants regard as their invention to recite a chemical mechanical polishing unit for chemical mechanical polishing integrated circuits and a chemical mechanical polishing effluent discharge for discharging a wastewater stream containing byproduct polishing slurry containing copper ions at a level in the range of about 1-100 mg/l; and a carbon bed connected directly to the chemical mechanical polishing effluent discharge.

New Claims 21-28 have been added to more particularly point out and distinctly claim that which Applicants regard as their invention to recite a chemical mechanical polishing unit for chemical mechanical polishing integrated circuits and a chemical mechanical polishing effluent discharge for discharging a wastewater stream containing byproduct polishing slurry containing copper ions at a level in the range of about 1-100 mg/l; a wastewater feed connected directly from the chemical mechanical polishing unit to a carbon bed for a wastewater feed containing hydrogen peroxide and metal ions in solution to and through the carbon bed, wherein the wastewater feed contains solids sized in the range of about 0.01-1.0 micron in an amount higher than about 50 mg/l to form a carbon bed product stream; and means for

passing the carbon bed product stream from the carbon bed to an ion exchange unit for removing the metal ions from solution.

Support for the Amendment to Claim 12 is found in Applicants' specification as originally filed on page 15, lines 2-9, and original Claim 16.

Support for the new Claims 21 - 28 is found in Applicants' specification as originally filed and the original Claims as filed.

New Claim 29 has been added to more particularly point out and distinctly claim that which Applicants regard as their invention to recite a chemical mechanical polishing unit of integrated circuits and a carbon bed for receiving a byproduct polishing slurry wastewater feed directly from the chemical mechanical polishing of integrated circuits, the byproduct polishing slurry wastewater feed containing copper ions in solution at a level in the range of about 5-25 mg/l and hydrogen peroxide, to reduce the concentration of hydrogen peroxide and to form a carbon bed effluent product stream having concentration levels of hydrogen peroxide less than about 0.1 mg/l, wherein the byproduct polishing slurry wastewater feed further contains solids sized in the range of about 0.01-1.0 micron in an amount higher than about 500 mg/l; an ion exchange bed of cross-linked polystyrene resin having a bead size in the range of about 0.4 to

1.23 mm for receiving a carbon bed product stream from the carbon bed and further having a macroporous iminodiacetic functional group for removing the copper ions from solution; means for passing to the carbon bed a byproduct polishing slurry wastewater feed from the chemical mechanical polishing of integrated circuits, the wastewater feed containing copper ions in solution at a level in the range of about 5-25 mg/l, hydrogen peroxide, and solids sized in the range of about 0.01-1.0 micron in an amount higher than about 500 mg/l and withdrawing a carbon bed product stream containing copper ions; means for contacting copper ions in the carbon bed product stream with the cross-linked polystyrene resin in the ion exchange bed to attach the copper ions and form an environmentally clean water discharge product; and means for regenerating the polystyrene resin with hydrochloric acid.

Support for the new Claim 29 is found in Applicants' specification as originally filed and the original Claims as filed.

35 U.S.C. §102

Claims 12-16 stand rejected under 35 U.S.C. §102(b) as anticipated by Faylor et al. U.S. Patent No. 3,870,033 (hereinafter "Faylor").

Faylor involves process and apparatus for ultrapure water.

Faylor discloses a deionized pretreated supply water and passing a part of the stored water through a pure water recirculation loop.

Applicants' Claim 12 as amended requires a chemical mechanical polishing unit for chemical mechanical polishing integrated circuits, having a chemical mechanical polishing effluent discharge valve for a wastewater stream containing byproduct polishing slurry containing copper ions at a level in the range of about 1-100 mg/l; a carbon bed connected directly to the chemical mechanical polishing effluent discharge and for receiving wastewater feed containing metal ions in solution, wherein the wastewater feed contains solids sized in the range of about 0.01-1.0 μ m in an amount higher than about 50 mg/l; and an ion exchange unit connected directly to the carbon bed, for receiving a carbon bed product stream from the carbon bed and for removing the metal ions from solution, which is nowhere taught or suggested in Faylor.

Applicant's Claims 12-15, as amended, require apparatus for treating a wastewater feed containing metal ions in solution, which is nowhere taught or suggested in Faylor. On the contrary, Faylor discloses the ability to remove ions with an ion-exchange

resin with a pure water feed that has no suspended solids. Faylor nowhere mentions or uses the presence of high levels of solids. The difference is important because Applicants' invention as claimed provides for the passing of the solids-containing waste slurry without appreciable retention in the column and the removal of peroxide and copper occurring with the passage of solids (50 to > 2000 mg/l). High solids feeds are passed through packed columns without the solids impacting the performance of that column.

For the foregoing reasons, the rejection of Claims 12-16 under 35 U.S.C. §102(b) as anticipated by Faylor et al. U.S. Patent No. 3,870,033 is based on an insufficient reference and is respectfully requested to be withdrawn.

35 U.S.C. §103

Claims 17-19 stand rejected under 35 U.S.C. §103(a) as unpatentable over Faylor et al. U.S. Patent No. 3,870,033, (hereinafter "Faylor") in view of Katzakian U.S. Patent No. 3,928,192 (hereinafter "Katzakian").

Claims 17-19 are dependent on Claim 12 as amended.

Applicants' Claim 12 as amended requires a chemical mechanical polishing unit for chemical mechanical polishing integrated

circuits, having a chemical mechanical polishing effluent discharge valve for a wastewater stream containing byproduct polishing slurry containing copper ions at a level in the range of about 1-100 mg/l; a carbon bed connected directly to the chemical mechanical polishing effluent discharge and for receiving wastewater feed containing metal ions in solution, wherein the wastewater feed contains solids sized in the range of about 0.01-1.0 μ m in an amount higher than about 50 mg/l; and an ion exchange unit connected directly to the carbon bed, for receiving a carbon bed product stream from the carbon bed and for removing the metal ions from solution, which is nowhere taught or suggested in Faylor.

Applicants' invention as claimed requires novel apparatus for the removal of copper ions from wastewater solution containing copper ions passed directly from a chemical mechanical polishing unit directly to and through a carbon adsorption column, without prior micro-filtration or ultra-filtration - removal of silica, alumina slurry solids, to remove catalytically the hydrogen peroxide (H_2O_2) and then contacting the wastewater solution containing copper ions to attach the copper, with a chelating ion exchange resin of iminodiacetic functional group.

passing a part of the stored water through a pure water recirculation loop. Applicants' Claims require apparatus for treating a wastewater feed containing metal ions in solution, which is nowhere taught or suggested in Faylor. On the contrary, Faylor discloses the ability to remove ions with an ion-exchange resin with a pure water feed that has no suspended solids. Faylor nowhere mentions or uses the presence of high levels of solids. The difference is important because Applicants' invention as claimed provides means for the passing of the solids-containing waste slurry without appreciable retention in the column and the removal of peroxide and copper occurring with the passage of solids (50 to > 2000 mg/l). High solids feeds are passed through packed columns without the solids impacting the performance of that column.

Katzakian discloses water demineralizing using separate beds of <u>weakly</u> acidic cation exchange resin, <u>weakly</u> basic anion exchange resin, and a third column of weakly acidic cation exchange resin. A saline feed is passed through the system.

There is no motivation to combine the Faylor and Katzakian references.

Katzakian involves ultra pure water which is completely removed from a solid wastewater feed to a carbon column with solid in the wastewater.

Even assuming, but not granting or admitting that one would have combined the Faylor and Katzakian references, one would have used a weak acid and weak base resin which is fully different from Applicants' invention.

Even assuming, but not granting or admitting that one would have combined the Faylor and Katzakian references, one would not have come up with Applicants' invention as claimed of novel apparatus for the removal of copper ions by passing a wastewater solution directly from a chemical mechanical polishing unit containing copper ions to and through a carbon adsorption column, without prior micro-filtration or ultra-filtration removal of silica, alumina slurry solids, to remove catalytically the hydrogen peroxide $(\mathrm{H_2O_2})$ and then contacting the wastewater solution containing copper ions to attach the copper, with a chelating ion exchange resin of iminodiacetic functional group, except on the basis of reconstructive hindsight after having had the benefit of Applicants' full disclosure as set forth in the detailed description of Applicants' Specification.

Applicants' invention as claimed requires means for passing the solids-containing waste slurry without appreciable retention in the column and for removing peroxide and copper occurring with the passage of solids (50 to > 2000 mg/l). A <u>high solids</u> feed is passed through packed columns without the solids impacting the performance of that column. This is not intuitively obvious and has not been for many years. Further evidence of the non-obviousness of Applicants' invention as claimed is observed in the feed specification published by the ion exchange resin manufacturer, which specifies a critical feed quality limitation as having a turbidity of <5 NTU's or in some cases <1 NTU. A chemical mechanical polishing CMP wastewater slurry is opaque, not transmitting to light, and infinitely outside the range of these feed specifications. Subsequent work in the field has been run with feeds having suspended solids in excess of 20% by weight (i.e. 200,000 mg/1).

Faylor discloses the ability to remove ions with ion-exchange resin with a pure water feed that has no suspended solids. This is obvious and the presence of high levels of solids is not addressed.

Faylor is significantly different from Applicants' invention as claimed. Faylor uses a variety of filtration devices, i.e.,

diatomaceous filters and/or pretreatment steps, i.e., alum/soda coagulation/flocculation followed by sand filtration to remove solids prior to carbon filtration. Applicants' invention as claimed differs significantly in that Applicants' invention does not remove suspended solids ahead of the carbon filter/ion exchange process for removing heavy metals. Applicants' invention as claimed has the distinct advantage in high solids loading applications such as this, of letting the solids which are largely non-regulated solids pass through the oxidant removal steps and through the ion exchange step. The copper, a commonly regulated metal, is removed in the ion exchange system, where it can be regenerated to form a concentrated copper solution that is recoverable by electrolytic recovery process or by specialized precipitation processes which precipitate the copper in a form suitable for resale. The copper-free solids then can be discharged to a sewer, if the suspended solids levels of this stream when combined with the remainder of the plant's effluent are below regulatory limits.

Alternately, the copper-free solids can be removed by coagulation and settling, direct filtration, and disposed of according to appropriate regulations.

Applicants' invention as claimed provides novel apparatus for the removal of copper ions from a byproduct polishing slurry wastewater solution containing copper from the chemical mechanical polishing (CMP) of integrated circuits of semiconductor microelectronic chips. The chemical mechanical polishing (CMP) planarization process introduces copper into the process water, and governmental regulations for the discharge of wastewater from the chemical mechanical polishing (CMP) planarization process are as stringent as the wastewater from an electroplating process, even though CMP planarization is not an electroplating process. The copper ions in solution in the wastewater must be removed from the byproduct polishing slurry for acceptable wastewater disposal. The chemical mechanical polishing planarization of the microchip produces a byproduct "grinding" (polishing) slurry wastewater which contains copper ions at a level of about 1-100 mg/l. The byproduct polishing slurry wastewater from the planarization of the microchip also contains solids sized at about 0.01-1.0 μm at a level of about 500-2000 mg/l (500-2000 ppm). An oxidizer of hydrogen peroxide (H_2O_2) typically is used to help dissolve the copper from the microchip. Accordingly, hydrogen peroxide (H_2O_2) at a level of about 300 ppm and higher also is present in the byproduct polishing slurry wastewater.

chelating agent such as citric acid or ammonia also can be present in the byproduct polishing slurry to facilitate keeping the copper in solution. If hydrogen peroxide (H_2O_2) is present, the ion exchange resin will be oxidized, and the resin structure is broken down. Accordingly, hydrogen peroxide can not be present in an ion exchange unit operation because the ion exchange resin is incompatible with hydrogen. Ion exchange can be used to attach copper ions, but would not be likely to work on a byproduct polishing slurry because of the quantities of solids coming in with the byproduct polishing slurry in the form of a silica, alumina slurry. Conventional pretreatment practice for granular activated carbon beds principally requires the removal of contaminants such as excess amounts of suspended solids. Suspended solids, including bacteria, in amounts exceeding about 50 mg/l (50 ppm), and commonly in amounts exceeding about 5 mg/l $\,$ (5 ppm) are required to be removed prior to operating the carbon Ion exchange resin suppliers and equipment manufacturers strongly advise that particle controls ahead of, i.e., upstream from, ion exchange bed systems are an essential aspect of an effective pretreatment system. Particles of suspended solids bind up the ion exchange resin beds. The resin acts as a filter to retain the particles. The suspended solids accumulate and

cause an increase in pressure drop across the resin bed. this increased pressure drop occurs, the water is forced to take the path of least resistance and circumvents or flows around the resin bed. This resin circumvention is called channeling. the process water flows down the sides of the column, a large portion of the resin is bypassed, limiting the contact between the resin and the process water, resulting in high contaminant leakage and poor bed capacity. Under extreme conditions, internal distributors and collectors can break due to the high pressure drop. An ion exchange bed that is loaded with solids is difficult to regenerate. The regenerant solution takes the path of least resistance and channels down the sides of the column resulting in incomplete regeneration of the resin. According to Rohm and Haas, the feed must be relatively free from suspended and colloidal material. The suspended and colloidal matter will form a mat at the surface of the bed. Pressure drop increases, channeling is encountered, and portions of the bed are by-passed. The suspended and colloidal matter also coat the beads and granules of the ion exchange resin, reducing the rate of diffusion of ions in and out of the exchanger resin. It is therefore important that all feeds be clarified as much as possible to remove the last traces of suspended or colloidal matter. Coagu-

lation, sedimentation, and filtration are the normal clarifying methods. The byproduct polishing slurry wastewater containing copper ions from the CMP of semiconductor microelectronic chips containing copper can be passed through a microfilter to remove the solids in the form of the silica, alumina slurry. The permeate from the microfilter containing permeate copper ions then can be contacted with the ion exchange resin to attach the copper. Further according to Rohm and Haas, pretreatment of the feed also should remove or neutralize traces of soluble constituents that may degrade or foul the exchanger, e.g., traces of oxygen, ozone, chlorine, and other oxidants. Wastewaters from non-copper CMP processes are generally discharged to the semiconductor fabrication facility end-of-pipe where the wastewater is neutralized prior to discharge. With the advent of copper technology, these slurry wastewaters will contain copper. Copper present in the fabrication facility outfall poses problems. fabrication facilities must control the amount of suspended solids in the out fall. Accumulation in the receiving POTW's (Publicly Owned Treatment Works) sludges result in increased cost for municipal sludge disposal and environmental concerns to eliminate copper in the municipal sludge. Bio-toxicity problems in the municipal biological systems are caused by mass loading of

copper. Environmental discharge limits for copper result in noncompliance at the fabrication facility.

Applicants' apparatus as claimed overcomes all of these problems and removes the copper from the waste slurries near the point of generation and permit a copper-free waste to pass to discharge and neutralization. Applicants' apparatus as claimed overcomes all of these problems and removes copper ions from solution for acceptable wastewater disposal of byproduct polishing slurries containing high amounts of suspended solids and removes the copper ions from solution containing high amounts of suspended solids efficiently and economically.

For the foregoing reasons, the rejection of Claims 17-19 under 35 U.S.C. §103(a) as unpatentable over Faylor et al. U.S. Patent No. 3,870,033 in view of Katzakian U.S. Patent No. 3,928,192 is based on an improper combination of insufficient references and is respectfully requested to be withdrawn.

Applicants earnestly request entry of this amendment after final as presenting rejected claims in better form for consideration on appeal.

Attached hereto is a marked-up version of the changes made to the Abstract by the current Amendment. The attached pages are captioned "VERSION WITH MARKINGS TO SHOW CHANGES MADE."

Reconsideration of this application is requested.

Respectfully submitted,

Attothey of Record

Reg. No. 29,640

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Serial No. 10/017,077 Group Art Unit 1724 VERSION WITH MARKINGS TO SHOW CHANGES MADE IN THE SPECIFICATION On page 1, on the first line after the title, insert -- This patent application is a Division of prior, co-pending U.S. Patent Application Serial No.: 09/113,982, filed July 10, 1998, now U.S. Patent No. 6,346,195.--. IN THE CLAIMS 12. (Twice Amended) Apparatus for removing metal ions from wastewater, comprising: (a) a chemical mechanical polishing unit for chemical mechanical polishing integrated circuits, said chemical mechanical polishing unit having a chemical mechanical polishing effluent discharge for discharging a wastewater stream containing byproduct polishing slurry containing copper ions at a level in the range of about 1-100 mg/l; (b) a carbon bed connected directly to said chemical mechanical polishing effluent discharge, said carbon bed providing means for receiving [a] said wastewater feed containing -24Serial No. 10/017,077 Group Art Unit 1724 metal ions in solution, wherein said wastewater feed contains solids sized in the range of about 0.01-1.0 μm in an amount higher than about 50 mg/l; and (c) an ion exchange unit connected directly to said carbon bed, for receiving a carbon bed product stream from said carbon bed and for removing said metal ions from solution. 21. Apparatus for removing metal ions from wastewater, comprising: (a) a chemical mechanical polishing unit for chemical mechanical polishing integrated circuits, said chemical mechanical polishing unit having a chemical mechanical polishing effluent discharge for discharging a wastewater stream containing byproduct polishing slurry containing copper ions at a level in the range of about 1-100 mg/l; (b) means for passing a wastewater feed directly from said chemical mechanical polishing effluent discharge to a carbon bed, said wastewater feed containing hydrogen peroxide and metal ions in solution through said carbon bed, wherein said wastewater feed contains solids sized in the range of about 0.01-1.0 micron in an amount higher than about 50 mg/l to form a carbon bed product stream; and -25Serial No. 10/017,077 Group Art Unit 1724 (c) means for passing said carbon bed product stream from said carbon bed to an ion exchange unit for removing said metal ions from solution. 22. Apparatus for removing metal ions from wastewater as set forth in claim 21, wherein said wastewater feed contains solids in an amount higher than about 100 mg/l. 23. Apparatus for removing metal ions from wastewater as set forth in claim 21, wherein said wastewater feed contains solids in an amount in the range of about 500-2000 mg/l. 24. Apparatus for removing metal ions from wastewater as set forth in claim 21, wherein said means for passing said wastewater feed containing hydrogen peroxide and metal ions in solution to said carbon bed reduces the concentration of said hydrogen peroxide and forms a carbon bed effluent having concentration <u>levels</u> of <u>hydrogen</u> <u>peroxide</u> <u>less</u> <u>than</u> <u>about</u> <u>0.1</u> <u>mg/l.</u> 25. Apparatus for removing metal ions from wastewater as set forth in claim 24, wherein said metal ions comprise copper ions at a level in the range of about 1-100 mg/l. -26-

26. Apparatus for removing metal ions from wastewater as set forth in claim 25, wherein said means for passing said carbon bed product stream from said carbon bed to an ion exchange unit comprises contacting metal ions in said carbon bed product stream with a resin having a macroporous iminodiacetic functional group.

27. Apparatus for removing metal ions from wastewater as set forth in claim 25, wherein said means for passing said carbon bed product stream from said carbon bed to an ion exchange unit comprises means for contacting said carbon bed product stream metal ions with cross-linked polystyrene resin to attach said copper ions.

28. Apparatus for removing metal ions from wastewater as set forth in claim 27, wherein said means for passing said carbon bed product stream from said carbon bed to an ion exchange unit comprises means for contacting said carbon bed product stream metal ions with cross-linked polystyrene resin screened to provide a bead size in the range of about 0.4 to 1.23 mm with a tight uniformity coefficient of about 1.7 to attach said copper ions.

Serial No. 10/017,077 Group Art Unit 1724 29. Apparatus for removing copper ions in a byproduct polishing slurry wastewater from the chemical mechanical polishing of integrated circuits, comprising: (a) a chemical mechanical polishing unit for polishing integrated circuits. (b) an carbon bed for receiving a byproduct polishing slurry wastewater feed directly from said chemical mechanical polishing of integrated circuits, said byproduct polishing slurry wastewater feed containing copper ions in solution at a level in the range of about 5-25 mg/l and hydrogen peroxide to reduce the concentration of said hydrogen peroxide and form a carbon bed effluent product stream having concentration levels of hydrogen peroxide less than about 0.1 mg/l, wherein said byproduct polishing slurry wastewater feed further contains solids sized in the range of about 0.01-1.0 micron in an amount higher than about 500 mq/l;(c) means for providing a ion exchange bed of cross-linked polystyrene resin having a bead size in the range of about 0.4 to 1.23 mm for receiving a carbon bed product stream from said carbon bed and further having a macroporous iminodiacetic functional group for removing said copper ions from solution; -28-

(d) means for passing to said carbon bed a byproduct polishing slurry wastewater feed directly from the chemical mechanical polishing of integrated circuits, said wastewater feed containing copper ions in solution at a level in the range of about 5-25 mg/l, hydrogen peroxide, and solids sized in the range of about 0.01-1.0 micron in an amount higher than about 500 mg/l and withdrawing a carbon bed product stream containing copper ions;

(e) means for contacting copper ions in said carbon bed product stream with said cross-linked polystyrene resin in said ion exchange bed to attach said copper ions and form an environmentally clean water discharge product; and

(f) means for regenerating said polystyrene resin with hydrochloric acid.